CHAPTER 2

EXPERIMENTAL PROCEDURE

2.1. Chemical reagents and equipments

2.1.1 Chemical reagents

1) Ammonium Molybdate, $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$, MW = 1235.86, Fisher Scientific, UK.

2) Silver Nitrate, $AgNO_3$, MW = 169.87, Poch, Poland.

3) Nitric acid, HNO_3, MW = 63.09, min 65.%, RCI Labscan, Thailand.

4) Sulfuric acid, H_2SO_4, MW = 98.08, min 96%, Carlo Erba, Italy.

5) Hydrochloric acid, HCl, MW = 36.46, min 37%, Carlo Erba, Italy.

6) Propylene glycol, $C_3H_8O_2$, MW = 76.09.

2.1.2 Equipment

1) Hotplate & magnetic stirrer, model 502P-2, PMC Industries, Inc., San Diego, America

2) Analytical balance, model BP-210S, Sartorius AG. Goettingen, Germany

3) Oven, model UE-400, Memmert, Germany

4) X-ray Diffraction, XRD, Philips X'Pert MPD, PANalytical B.V., Netherlands

5) Field emission scanning electron microscope, SEM-FE, JSM-6335F, JEOL, Japan

6) Low vacuum scanning electron microscopy, LV-SEM, JEOL, JSM-5910LV, Japan

7) Transmission electron microscope, TEM, JEM-2010. JEOL, Japan

8) Fourier transform infrared spectroscopy, FTIR, TENSER 27, BRUKER, USA

9) Raman spectroscopy, T64000, HORIBA JOBIN YVON, Japan

10) UV-Visible spectrometer, Varian Cary 50 Scan, Varian, Inc., America

11) Luminescence spectrometer, LS50B, Perkin Elmer, America

2.2 Synthesized methods

2.2.1 Synthesis of Molybdenum Oxide by a Hydrothermal method

The first, 0.005 mole of ammonium heptamolybdate tetrahydrate ($(NH_4)_6Mo_7O_{24}.4H_2O$) were dissolved in 20 ml deionized water with continuous stirring at room temperature for 30 min. Then 15 ml 2M HNO_3 solution was added in that colorless solution and stirred for 30 min. It was transferred into a Teflon-lined stainless steel autoclave to a capacity volume of 50 ml. The autoclave was tightly closed and heated at 180 °C for 20 h in the electric oven. At the completion of the reaction, light-blue precipitates were separated by filtration, washed with distilled water to remove ions possibly remaining in the final products and ethanol, and finally dried at 80 °C in an electric oven for 24 h. And the effects of reaction temperature (100-180 °C for 20 h), holding time (200 °C for2-20 h) and difference

types of acid (HCl and H_2SO_4) were studied on the formation of α-MoO_3 nanobelts. Experimental conditions for synthesizing of MoO_3 is shown in Table 2.1

Table 2.1 Experimental conditions for synthesizing of MoO_3

Mo source	Acid	Reaction Temp.(°C)	Holding time(h)
$(NH_4)_6Mo_7O_{24}.4H_2O$	HNO_3	180	20
$(NH_4)_6Mo_7O_{24}.4H_2O$	HNO_3	120	20
$(NH_4)_6Mo_7O_{24}.4H_2O$	HNO_3	140	20
$(NH_4)_6Mo_7O_{24}.4H_2O$	HNO_3	160	20
$(NH_4)_6Mo_7O_{24}.4H_2O$	HNO_3	180	2
$(NH_4)_6Mo_7O_{24}.4H_2O$	HNO_3	180	5
$(NH_4)_6Mo_7O_{24}.4H_2O$	HNO_3	180	10
$(NH_4)_6Mo_7O_{24}.4H_2O$	H_2SO_4	180	20
$(NH_4)_6Mo_7O_{24}.4H_2O$	HCl	180	20

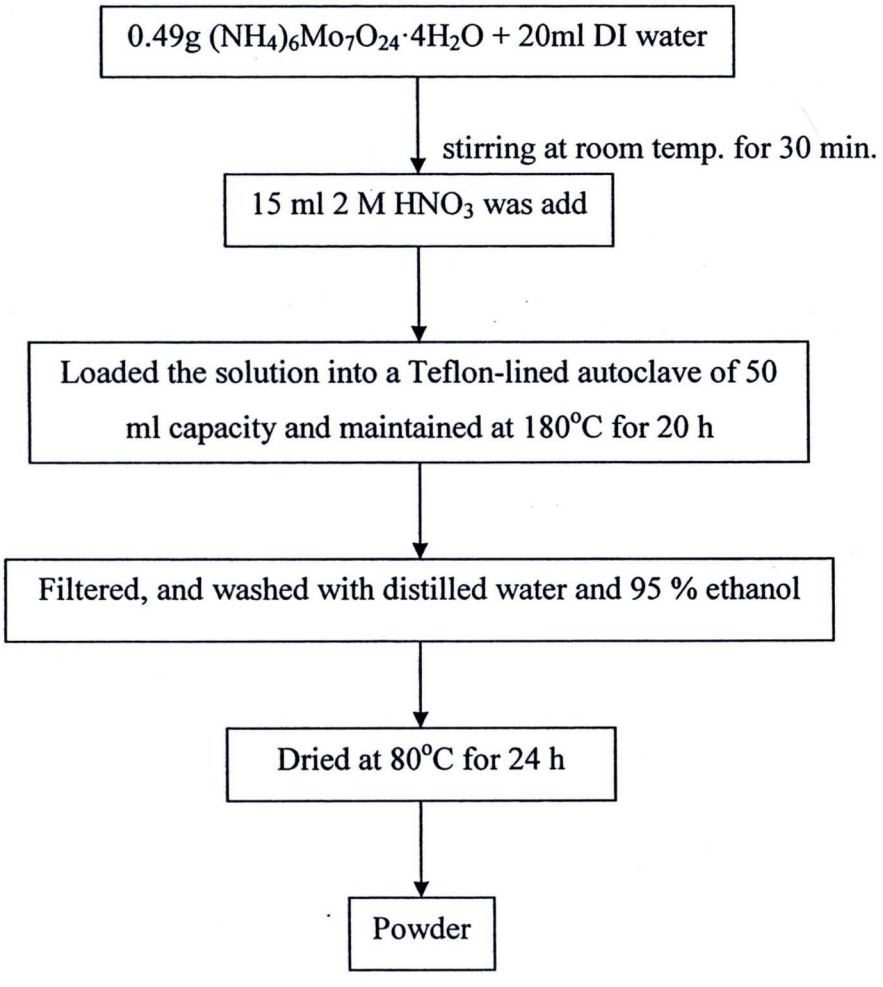

Figure 2.1 Schematic diagram for the synthesis procedure of the MoO_3 using Hydrothermal method.

2.2.2 Synthesis of Silver composite on Molybdenum Oxide nanobelts by a Sonochemical method

The solutions were prepared by dissolving 1.0, 2.5, 5.0, 7.5, 10.0 wt.% Ag on α-MoO_3 nanobelts(gram of $AgNO_3$ and MoO_3 show in table 2.2) and total weight of 1.5 g in 50 ml Propylene Glycol. Ultrasonic irradiation was maintained at room temperature for 20 min. and operated at a frequency of approximately 50-60 kHz at 220 W output power. The resulting light-blue materials were washed with water and ethanol, and dried at 80°C for 24 h.

Table 2.2 $AgNO_3$ and MoO_3 used for the synthesis of Silver composite on Molybdenum Oxide nanobelts

%Ag	MoO_3(g)	Ag(mol)	$AgNO_3$(g)
1.0	1.4850	0.00014	0.0238
2.5	1.4625	0.00035	0.0591
5.0	1.4250	0.00069	0.1172
7.5	1.3875	0.00104	0.1767
10.0	1.3500	0.00140	0.2378

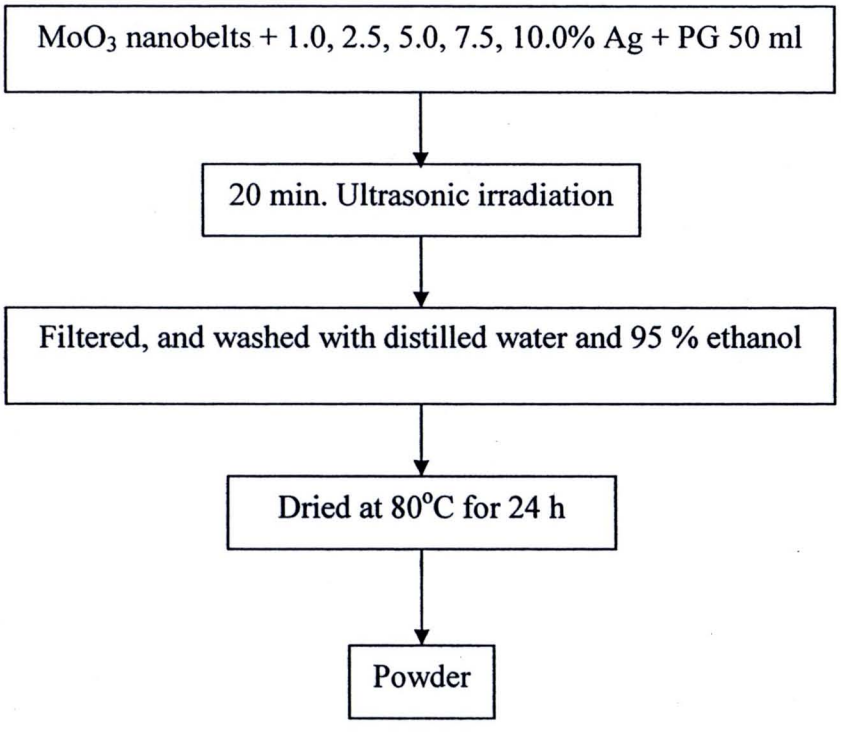

Figure 2.2 Schematic diagram for synthesis Silver composite on Molybdenum Oxide nanobelts by a Sonochemical method

2.3 Characterization

2.3.1 X-ray Diffraction

The crystallinity and phase purity of the products were analyzed by using X-ray diffractometer (XRD) with Cu K_α radiation (λ = 1.5418 Å) operating at 20 kV- 15mA, at a scanning rate of 5°/min in the 2θ range of 10°-60°. The phase was identified by Philips X'Pert Highscore Computer Software (search-match program) on the database of JCPDS software.

Figure 2.3 X-ray Diffractometer

2.3.2 FT-IR and Raman Spectrometry

Fourier transform infrared spectroscopy with KBr pellet technique and operated in the range of 400–4000 cm^{-1}. Raman spectrometer Triple monochromator with Ar laser 514.32 nm and 50 mW by Jobin Yvon Horiba was used.

Figure 2.4 Fourier transform infrared spectrometer

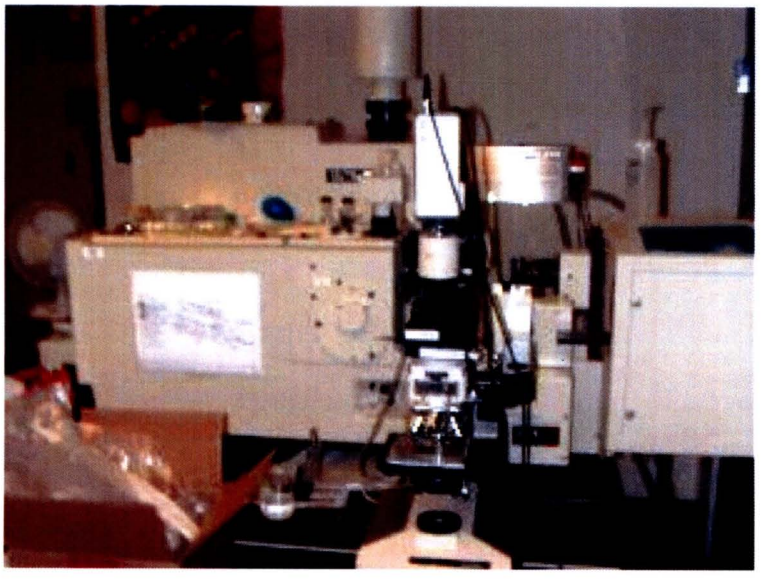

Figure 2.5 Raman spectrometer

2.3.3 Scanning electron microscopy

The morphology and particle sizes of as-synthesized samples were determined by a scanning electron microscope operated at 15 kV accelerating voltage. The chemical composition was investigated by energy dispersive x-ray (EDX) analyzer equipped to SEM and controlled by Inca program used to determine the chemical composition.

Figure 2.6 Scanning electron microscope

2.3.4 Transmission electron microscopy

The particle size and morphology were characterized by transmission electron microscope operating at 20 kV. The samples for TEM analysis were prepared by dispersing small amount of the powder in absolute ethanol and placing a drop of the solution onto a copper grid coated with holey carbon film and leaving the ethanol evaporate slowly in air.

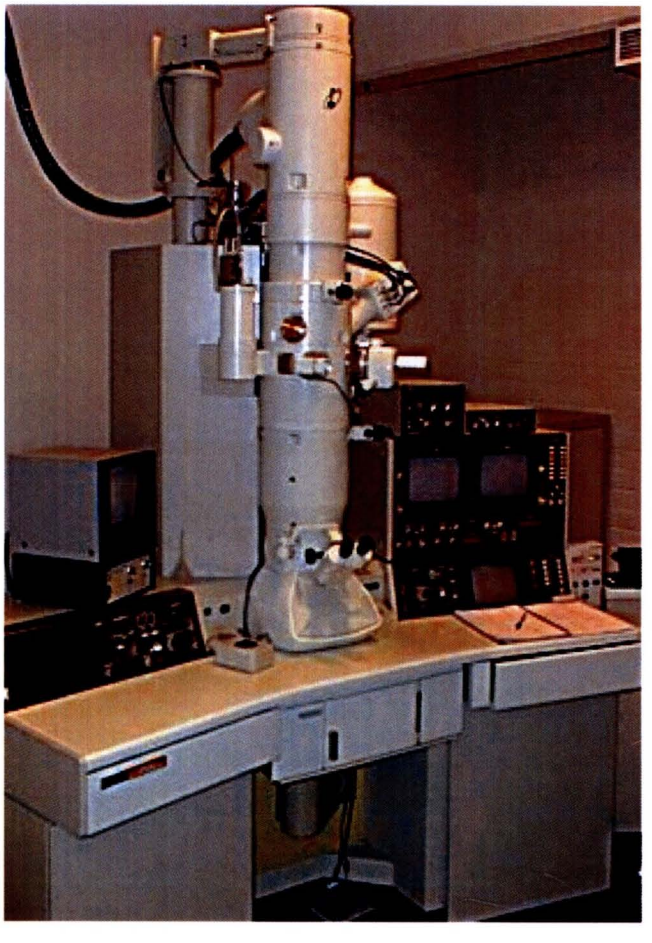

Figure 2.7 Transmission electron microscope

2.3.5 UV-Visible spectrometry

UV-Visible spectrometry is a general technique to point out correlations between spectra and structure to be used. The spectrum obtained directly from an instrument is simply a plot of wavelength (or frequency) of absorption versus the absorption intensity (absorbance or transmittance).

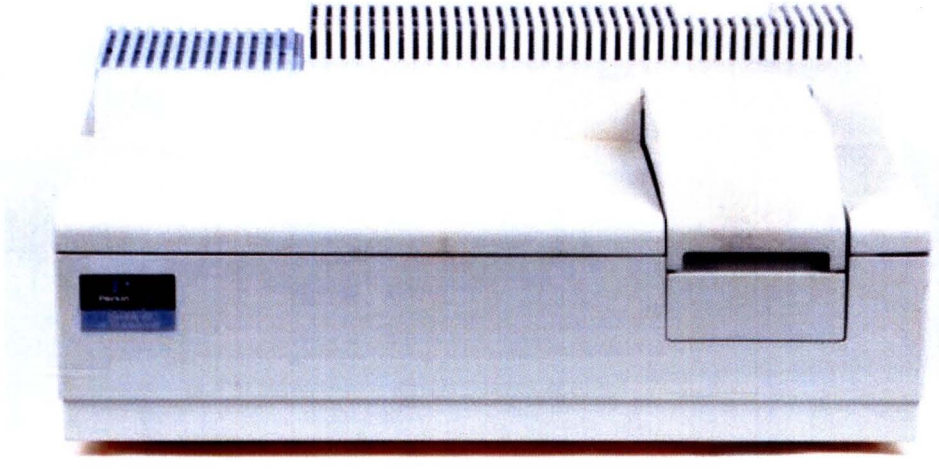

Figure 2.8 UV-visible spectrophotometer

2.3.6 Luminescence spectrometry

The luminescence emission spectra of the samples were investigated using luminescence spectrometer at room temperature. The appropriated amount of powder samples were dispersed in absolute ethanol using ultrasonic bath, and test for emission.

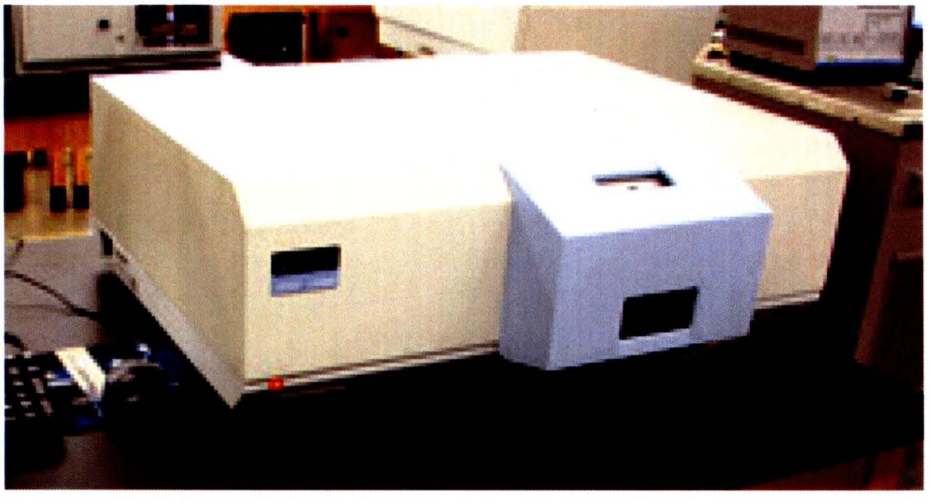

Figure 2.9 Luminescence spectrometer